Measurement of batch properties

5   The present invention relates to the measurement of
bulk properties of products produced in batches. It is
particularly, but not exclusively applicable to products
formed as fluids or particles. One useful application of
the invention concerns the batch production of polymers.

10   Polymer production plants produce large quantities
of polymer, commonly by means of the continuous Borstar
or Phillips processes using loop or gas phase reactors.
The raw materials such as monomer, comonomer, catalyst,
diluent etc. are supplied to the loop reactor where they

15   are circulated in the form of a slurry. The reactor is
maintained under high pressure so that the monomer gas
is maintained in liquid form.

           In some processes the polymer forms as solid
particles of polymer fluff. These are allowed to

20   precipitate out of the slurry in so-called settling legs
from which the concentrated slurry is periodically
discharged. The solid matter is separated from the
diluent in flash tanks where the diluent is allowed to
vaporise before being recycled.

25   The solid is then transported from the reactor
entrained in gas in a pneumatic system. In order to
produce a product that is in a convenient form for
transport to customers, and in order to stabilize the
product, the polymer fluff is fed to an extruder in

30   which it is melted, mixed with additives, homogenized
and formed into pellets. The pellets are then fed to
large silos containing about 70 to 500 tonnes or more of
product. Each silo of product comprises a single batch.

It will be appreciated that the control of such a process is highly complex; sophisticated computer-based systems are often used to do this. There are a great number of factors that have an effect on properties of

5   the finished product. For example, the reactor conditions and catalyst properties determine the size of polymer molecules (i.e. the molecular weight), the molecular weight distribution (MWD) and the co-monomer incorporation, that in turn determine the melt flow rate

10 and density of the polymer.

Within each basic type of polymer such as polyethylene, polypropylene etc, products are classified by manufacturers into defined grades. These each have a set of specified properties that must be met within

15 given tolerances. Thus, a grade of polyethylene may be specified as having a certain MFR and a given density.

It follows that in order to produce a given grade of polymer the critical product properties must remain substantially consistent. However, it is inevitable that

20 during a production campaign there will be some significant variation in the instantaneous value of the various parameters concerned. This is not, in itself, a serious problem because these properties will, to some extent, average out within the large volume that forms a

25 batch of production. A small degree of product variation can therefore be tolerated provided that it does not put the bulk property of the batch out of specification.

The conventional way of checking the bulk properties of a batch of polymer is to "blend" the batch

30 (i.e. to mix it thoroughly) and then to take one or more small samples. These are taken to be representative of the bulk properties of the polymer. The samples are then taken away for laboratory analysis in order to check

whether they (and therefore also the batch of polymer)
are within the specification for the particular grade.

It will be appreciated that this method is very
time-consuming and in many cases is too slow to allow
any remedial action to be made to the production
process. Consequently, it may be discovered too late
that the production is not usable as the desired grade.
There are also problems with the reliability of any such
sampling technique as it relates to what is inevitably a
tiny proportion of a, for example, 150 tonne batch.

More recently, useful online measurement techniques
have become available. These enable product properties
to be measured or determined in almost real time. Thus,
on the basis of such measurements, it is possible to
monitor the polymer product as is produced. If the
product is found to be out of specification, remedial
action can be taken by modifying process conditions to
bring future production back onto specification.

It will be appreciated that this provides a
significant advantage over the earlier technique.
However, the inventors have recognized that such a
system has a significant drawback: whilst it can prevent
further out of specification product from being made, it
does not give sufficient information about the effect of
the product already made on the bulk properties of the
finished product that is already in the silo. The grade
of the finished product must still be checked by
sampling, as before. Furthermore, it is not possible to
remedy the effect of the out of specification
production. There are therefore previously unrecognized
and significant problems with prior art quality-control
techniques.

Viewed from one aspect the present invention

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provides a method of monitoring a bulk property of a
product during its production comprising the steps of
of:

   a) making repeated on-line measurements of samples
5   of the product to obtain data related to a
         product property; and

   b) using this data, determining a bulk property of
      the product so far produced.

As mentioned above, and as will be discussed more
10 fully below, the invention is applicable to the
production of a wide range of materials, especially
those formed as fluids or particles. The field of
polymer production is one example, but there are many
others such as gas production, drinks, powders, etc.

15   The bulk property may directly correspond to the
product property of step (a). For example, if sample
density is measured in step (a) then the bulk property
could be the overall density of the batch of product. It
may, however, be a property that is derived from the
20 data obtained in step (a) but which is never obtained in
respect of the individual samples. An example of this
would be a measure of distribution or spread such as the
standard deviation of the molecular weight.

Thus, by means of the invention, it is possible to
25 check the grade of a batch of product immediately the
batch is complete. There is no need to blend the product
and then to take small samples for laboratory analysis.
This saves a significant amount of time, and therefore
reduces costs compared to the prior art sampling
30 .technique. Furthermore, the bulk property data obtained
according to the invention is likely to be much more
accurate and representative of the product as a whole
than the small samples used in the prior art technique.

It is not necessary to wait until the end of
production to determine the bulk property; the invention
may be used to provide such information about the
product produced so far at any stage of production.

5   Indeed, it is particularly preferred that the invention
be used to provide repeated or continuous monitoring of
a property or properties throughout the production of
the batch.

It will be seen that this preferred form of the

10   invention is particularly useful because bulk properties
can now be checked during production so there can be
confidence that a batch of product is going to be on-
specification.

A further significant benefit is provided in

15   accordance with a preferred form of the invention in
which the data produced in step (b) of the invention is
used to assist in controlling the production plant. It
will be appreciated that instead of simply bringing
current production back into specification, using the

20   invention in this manner allows a correction to be made
to compensate for previously out of specification
product.

Thus, a significant benefit is provided in that a
product that would otherwise be out of specification

25   (which would therefore have had to be either wasted or
sold as a lower grade) can be remedied and brought back
within the specification of the desired higher grade.

The invention may be used to assist in the manual
control of the production plant. For example, a display

30   may be provided in the control room indicating the   ,
current bulk property (e.g. bulk density of polymer
contained in a silo), preferably together with the
current sample density (i.e. the density of the current

production) and the target bulk density. The plant
controller can then take remedial action when necessary.
Thus, if the bulk property has drifted away from
specification, the current properties of the production

5   may be varied (possibly putting them temporarily out of
specification) in order to bring the bulk property
closer to the desired bulk specification.

As an example, if the calculated overall density of
the product were too low, for a limited period, product

10   could be made at higher than specification density in
order to bring the bulk density back to the desired
value.

There may also, for example, be cases where the
instantaneous property is too high (according to

15   specifications), but the calculated batch property up to
this specific time shows that it will be on the low side
of the specification. Thus the correct intervention from
the operator is to keep the property at its current
(high) level (if the goal is to get as close as possible

20   to the specification target).

It will be appreciated that in many circumstances
there may be a limit to how far such corrections can be
made without making the standard deviation of the
property excessively large (which may itself put the

25   product out of specification). This should be considered
when determining how frequently the on-line measurements
should be made. If the measurements are made on a
frequent basis then the corrections involved should be
comparatively small. In fact, a sample having an

30   excessively large standard deviation would generally not
be detected by the known (spot sampling) technique,
whereas it may readily be detected by means of the
present invention.

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Thus, it will be appreciated that the repeated
measurements should preferably be made sufficiently
frequently to follow significant fluctuations in product
quality. In most cases, this means that a measurement
5   should be taken at least every 10 minutes and preferably
more frequently than this, for example at least every 5
minutes. However, if a product quality is liable to
fluctuate rapidly then intervals of less than two
minutes, and possibly even as short as one minute, may
10   be appropriate.

Although suitable time intervals may be determined
empirically, it is preferred that the sampling frequency
be calculated using the well-known Nyquist Sampling
Theorum (see H. Nyquist, "Certain Factors Affecting
15   Telegraph Speed", The Bell System Technical Journal,
Vol. 3, pp. 324-47, July 1924.) This may be stated as:
*"If the signal we are looking at changes at a maximum
frequency of f, then we must sample at least 2*f to
capture the detail"*. Thus, preferably the sampling
20   frequency is at least twice the frequency of anticipated
significant changes in the product property being
tested.

Although, as mentioned above, the data may be used
manually by a plant controller, it is preferred that the
25   correction process be automated. Thus, the method of
the invention may be performed under computer control
and be linked to an automatic process control system. To
achieve this, the on-line measuring device may be
arranged to provide an output signal that is fed, via an
30   analogue to digital converter to an input port on the
computer. At predetermined times, under software
control, the input may be read and its value used to
determine a value corresponding to the bulk property by

means of a suitable software routine. This value may then be used to provide an output signal that may in turn be fed to an automatic process controller.

Thus, viewed from another aspect, the invention provides a method of controlling a production process in which data directly related the aggregate properties of the batch of product produced so far are used to control the process in order to maintain the aggregate properties within specification.

As previously noted, the invention is widely applicable. Although the present specification discusses the invention in detail in relation to polymer production, there are numerous other fields of application, particularly in relation to gaseous, liquid, powder and pellet (or other particle) production.

By way of example, the invention may be applied in the production of oxygen for clinical use, which has very strict limits on purity. By measuring the purity with an online instrument, it is possible to calculate the purity of a batch (stored in a pressurised vessel, possibly liquefied). The purpose of the online measurement is thus twofold: to monitor the production process, and to calculate the purity of the batch. The fluid (oxygen and impurities) will be homogenous throughout the tank after some time (whether it is liquefied or not), and any fraction bottled from the tank will have the purity calculated by the method described.

As a further example, the invention may also be applied in a similar (although less critical) manner in the production of soft drinks. These are commonly produced in batches and it is desirable for each batch

to be produced to a similar specification. Thus, online
measurement of the product e.g. as it is fed to a
storage vessel may be used to calculate the properties
of the bulk product within that vessel.

5   Any sort of online measurement that produces data
that gives information relating to any useful batch
property may be used in the invention.

For example, particularly in the field of polymer
production, a spectrometer, such as an acoustic
10   spectrometer or a spectrophotometer may be employed. For
example, an NIR (near infra-red) spectrophotometer may
be used to measure the spectrum of polymer fluff passing
through a conduit from the production plant. As is well
known in this art, such apparatus may be used to provide
15   information from which product density may be derived.
The set of repeated samples of this data may then be
used to derive the density of the batch of polymer
produced so far.

Another option is to use rheometric measurements.
20   For example, a rheometer may be associated with an
extruder which is used to homogenise and pelletise the
polymer fluff. In such an apparatus, viscosity
measurements are made at various pressures and these may
in turn be used to determine the melt flow rate which is
25   related to the molecular weight of the polymer. They
corresponding bulk property can then be calculated.

Where the property concerned is additive, samples
are taken at regular intervals and the production rate
is substantially constant, it may only be necessary to
30   determine the mean of the values of the property
determined in step (a) from the start of the batch
onwards. However, certain properties, such as density,
are not additive. (The overall density of a set of

particles mixed together is not equal to the mean of the
individual densities of the particles.) In such cases
more complex calculations are required – in the case of
density the bulk (reciprocal of density) may be found,
then averaged and converted into a density value.

However, it is common for production rate to vary
significantly, especially at the beginning or end of a
production campaign. Moreover, certain variations in
product property are related to changes in production
rate. Preferably, therefore, the calculation of a batch
property takes into account the production rate at the
times the relevant measurements occur. In this way the
values corresponding to high production rate can be
weighted accordingly.

This may be achieved by measuring the flow rate of
the product passing through the online measuring
apparatus if all the product passes via that apparatus,
or by measuring the flow rate separately if the online
measurement is taken on a bypass from the main conduit.
In this way, it may be determined what quantity of
product is produced with the particular measured value.
The flow rate may be measured using any suitable known
apparatus, such as a weight loss feeder.

However, as an alternative to directly measuring
the flow rate of product, it may be calculated, for
example on the basis of mass balance considerations.

Once the measured values of product properties and
the corresponding production rates are known, the bulk
property may be determined in various ways. Preferably,
the batch properties are calculated continuously through
the production time of a batch, and integrated with
respect to the production rate.

As has already been discussed, a simple case is

when the product property is additive. The integrated
property is then given by:-

$$P_{final} = \frac{\int_0^T \left(p_{(t)} \cdot \dot{m}_{(t)} \right) dt}{\int_0^T \dot{m}_{(t)} dt}$$

Where $p_{(t)}$ is the measured property value at time t, $\dot{m}_{(t)}$
is the measured production rate at time t(volume or mass
flowing through a given point per unit time), and T is
the total production time for the batch. If the property
is non-additive, an integral describing the mixing must
be used. The discrete form of the integral is then
found. For the above additive property integral, the
discrete form may be determined using the trapezoid
integral method.

In addition to this, the standard deviation (and/or
other parameters related to statistical process control
(SPC)) for all calculated property values throughout the
production time of the batch may be calculated. This may
be compared to the expected standard deviation for the
online measurement method (the measurement noise). If
the calculated standard deviation for the process is
higher than the expected standard deviation for the
measurement method, there may be property
inconsistencies in the batch. A final check could also
be to plot the distribution of all property
measurements, to detect bi- or multi-modal distributions
of a property.

As a more concrete example, consider the measured
property $p_{(t)}$ and the corresponding production rate $\dot{m}_{(t)}$
at times t during the production of a batch:

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$$
\begin{array}{c c c c c}
t & 0 & 1 & 2 & 3 \\
p_{(t)} & 3 & 4 & 4 & 1 \\
\dot{m}_{(t)} & 3 & 1 & 2 & 6
\end{array}
$$

Disregarding the variation in the production rate indicated by the different values of $\dot{m}_{(t)}$, the property mean for the batch is:-

$$
\overline{p} = \sum_{i=1}^{4} \frac{p_{(i)}}{n} = \frac{3+4+4+1}{4} = \underline{\underline{3}}
$$

However, using the trapezoid numerical integration method which does account for the changes in production rate gives:-

$$
p_{final} = \frac{\int_{0}^{T} \left(p_{(t)} \cdot \dot{m}_{(t)} \right) dt}{\int_{0}^{T} \dot{m}_{(t)} dt} = 2.6
$$

As a further example, density, which is not an additive property, is considered. Because it is non-additive, a special integral has to be developed. For polyolefins, specific volume $(1/\rho)$ is sufficiently additive to give accurate results. The integral is then:-

$$
\rho_{final} = \frac{\int_{0}^{T} \dot{m}_{(t)} dt}{\int_{0}^{T} \left(\frac{1}{\rho_{(t)}} \dot{m}_{(t)} \right) dt}
$$

Where $\dot{m}_{(t)}$ is mass flow rate. The expression in the denominator is then the volumetric rate. This integral may then be used as previously described.

The invention also extends to an apparatus which may be used to perform the method of the invention and so viewed from a further aspect the invention provides an apparatus for monitoring a bulk property of a product during its production comprising an input for receiving data corresponding to repeated on-line measurements of samples of the product which provide data related to a product property, the apparatus being arranged to use this data to determine a bulk property of the product so far produced.

Preferably the apparatus is also arranged to receive production rate data as previously discussed and to use such data in determining the bulk property.

Although such apparatus may be supplied separately from the source(s) of the input data, in use the apparatus further comprises one or more measuring devices such as those already described which supply the input data.

The determination of the bulk property is preferably carried out by means of a computer under software control. It may use an algorithm based upon the principles described above. The software may also be used to determine when input data is to be read.

An example of such an algorithm written in pseudo-programming language follows. It is suitable for use with an additive property, and finds both the accumulated mean of the property and the corresponding standard deviation:-

for each new property measurement p and corresponding instantaneous production rate m, do:

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```
        if first sample then
                -> Standard deviation std is zero when we
                -> only have one sample
                std = 0

 5
                -> Accumulated production n
                n = m

                -> Property mean pm
10              pm = p

                -> Sum of squares ss
                ss = m*p*p
        else
15              -> Find the accumulated production n
                n = n_(old) + m

                -> Update estimates on property mean pm
                pm  = ((n-m)*pm_(old) + m*p) / n
20
                -> Update estimates on sum of squares ss
                -> and standard deviation std:
                ss = ss_(old) + m*p*p
                std = sqrt((ss - n*pm*pm) / (n - 1))
25      end if

    end for
```

The sampling interval is constant. Production rate
30 m is the instantaneous production rate at the time of
the data aquistion. It is assumed that the production
rate during each cycle is constant. This is a valid
assumption when the time interval between each sample is
short.

35   It will be seen that accumulated production n is
updated each sampling interval by adding to it the
instantaneous production rate m. (As the sampling
interval is constant it is not necessary to convert rate
m into standard units). In addition, property mean pm is
40 repeatedly updated to provide a mean value of property
measurement p for the accumulated production. The new
value of pm is found by multiplying the old value of pm
by the old value of n (=n-m), adding this to the current
value of p multiplied by the current production rate m
45 and dividing the answer by the new accumulated

production n. Thus, it provides a suitably weighted mean
of pm (accumulated mean value of p. Likewise, an updated
value of the standard deviation (std) is found. As may
be seen, this uses an updated accumulated sum of the
current product property measurement p squared and
multiplied by m. From this is subtracted the updated
product mean pm squared multiplied by n (n being the sum
of all m). The result of this is divided by (n-1) and
the square root found to give the standard deviation.

It will be seen that the algorithm works on the
basis of continually updating the values of mean and
standard deviation, rather than calculating them afresh
each cycle. This is highly advantageous. As an example,
the additive property p was measured to be 2 at t1 and 3
at t2. The mass flow rate m was 7 kg/s at t_1 and 6 kg/s
at t_2, and the time distance between t_1 and t_2 was 1 sec.
Thus we have 7 mass units with property p=2 between t_0
and t_1, and 6 mass units with property p=3 between t_1 and
t_2. The property mean is:

$$\bar{p} = \frac{7 \cdot 2 + 6 \cdot 3}{7 + 6} = 2,46$$

The standard deviation is:

$$std = \sqrt{\frac{7 \cdot (2 - 2,46)^2 + 6 \cdot (3 - 2,46)^2}{7 + 6 - 1}} = 0,52$$

This way of calculation the mean and standard
deviation is cumbersome as one has to store all p and m
data for each new t until a batch is completed, and do a
complete recalculation at each·step. With the updating
method used in the above algorithm, this is not
necessary. The updating method is also very simple to
implement in any computer system.

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A further consideration is that, if there is a very large number of measurements, the sum of squares in the updating method will eventually become too large to process. If necessary, this problem may be overcome

5   using the mean sum of squares in the algorithm.

An alternative algorithm that uses trapezoid integration and which may be adapted for use with non-additive properties follows:-

```
10   for each new property measurement p and corresponding
     instantaneous production rate m, do:
             if first sample then
                     -> Standard deviation std is zero when we
                     -> only have one sample
15                   std = 0

                     -> set initial values for t_old n and m_old
                     t_old = 0
                     n = 0
20                   m_old = initial production rate (at t = 0)

                     -> Find the accumulated production n
                     -> (trapezoid integration):
                     n = n + (t - t_(old))*(m + m_(old))/2
25
                     -> Property mean pm (and helper variable pmprod)
                     pm = p
                     pmprod = p*m

30                   -> Sum of squares ss
                     ss = m*p*p
             else
                     -> Find the accumulated production n
                     -> (trapezoid integration):
35                   n = n + (t - t_(old))*(m + m_(old))/2

                     -> Update estimates on helper variable pmprod
                     -> and property mean pm (trapezoid integration):
                     pmprod = pmprod_(old) +
40                           (t - t_(old))*(p*m + p_(old)*m_(old))/2
                     pm  = pmprod / n

                     -> Update estimates on sum of squares ss
                     -> and standard deviation std:
45                   ss = ss(old) +
                          (t - t(old))*(m*p*p + m(old)*p(old)*p(old))/2
                     std = sqrt((ss - n*pm*pm) / (n - 1))
             end if

50   end for
```

It will be seen that this algorithm follows the

same basic structure as the one described earlier, but that since it uses trapezoid integration the time interval between each cycle may vary.

There may also be provided suitable display equipment and/or output devices to enable data to be transmitted to process control systems.

More generally, the apparatus is preferably configured to operate in accordance with some or all of the preferred aspects of the method of the invention previously described.

The invention further extends to a production facility for producing product in batches, such as a polymer production plant, that either uses the method of the invention or incorporates the apparatus of the invention. It also extends to product made by means of the invention.

Certain embodiments of the invention will now be described, by way of example only, with reference to the accompanying drawings: -

Figure 1 is a schematic diagram illustrating a typical polymer production plant in which the present invention may be incorporated;

Figure 2 of is a schematic diagram illustrating a polymer production plant incorporating a first embodiment of the invention; and

Figure 3 corresponds to Figure 2 but is modified in order to incorporate a second embodiment of the invention.

Figure 1 illustrates, in a highly schematic form, a typical polymer production plant. The main plant apparatus 1 comprises a source of reactants, catalysts etc 2 which are connected via and number of control valves (illustrated and collectively at 3) to loop

reactor 4.

Slurry containing a high concentration of polymer
fluff leaves reactor 4 via a settling leg (not shown)
from which it is fed to a flash drum 5. In the flash
5   drum 5, the polymer fluff is separated from the other
components which may be partly recycled.

Polymer from the flash drum is then carried
pneumatically along conduit 7 to extruder 8 where it is
melted, homogenized and turned into pellets.

10 The pellets are then transported via conduit 9 to
silo 10. The silo 10 typically has a capacity of around
150 metric tonnes which comprises a single batch of
production.

The production plant 1 is controlled by a
15 computerized automatic control system 6 which uses
various input measurements (not shown) on the basis of
which it controls the flow of reactants into the reactor
by a controlling valves 3. It also controls the reactor
conditions, etc.

20 It should be understood that such reactors are
extremely well known and so a highly simplified
description is given here is merely to place the
subsequent embodiments in context.

The first embodiment of the invention is
25 illustrated in Figure 2. It will be seen that production
plant 1, extruder 8, silo 10 and process controller 6
are as shown in Figure 1. To these components has been
added a weight loss feeder 11 which measures mass flow
rate, an NIR spectrometer 12 and data processor 13.

30 The basic principle of operation of the production
plant is as described in relation to figure 1.
As polymer produced by the plant 1 passes along conduit
7 the weight loss feeder 11 measures its mass flow rate.

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It is then passed through spectrometer 12 which
produces a near infra-red spectrum of the polymer. The
mass flow rate and the spectral data are transmitted to
data processor 13 where they are used to calculate

5   firstly the instantaneous polymer density and then the
bulk density of the polymer in the silo. This is done
using an algorithm based on the previously described
equation for bulk density. The output from a data
processor 13 is fed to process controller 6 which, if

10   necessary, makes suitable adjustments to process
conditions in order to maintain the desired bulk density
value for the product in the silo 10. Alternatively, or
additionally the data is presented in the plant control
room on a display. The integrated density is displayed

15   to the operator as an absolute number, and as a graph.
The number is useful to the operator in the sense that
the operator knows (at any time) what the final property
value for the complete batch is.

Figure 3 illustrates a second embodiment of the

20   invention. Again, the basic components of the plants are
unchanged. However, a mass-flow measuring device 21
corresponding to device 11 in Figure 2 is provided,
together with a rheometer 20 and a data processor 23.

A small proportion of polymer flowing through the

25   extruder is diverted through a bypass 22 which leads to
a rheometer 20. This produces data concerning the melt
flow rate of the polymer in the known manner. This data
is then transmitted, along with the mass flow data from
device 21 to data processor 23. The data processor 23

30   calculates the corresponding properties of the bulk
material in the silo in a manner directly analogous to
that described in respect of the first embodiment.